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A PROGRESS REPORT ON MELTING METHODS AND QUALITY LEVELS OF PMRL INGOTS

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A PROGRESS REPORT ON MELTING METHODS AND QUALITY LEVELS OF PMRL INGOTS

by

A.R. Palmer

SUMMARY

Clustered alumina inclusions were the main cause of poor ingot quality. Sulphide inclusions were of comparatively little significance since the steel normally contained less than 0.010% S.

Deoxidation and teeming procedures have been changed to eliminate the clustering of alumina inclusions. It was not possible however to reduce the total oxygen content to any measurable extent, because of the need to melt and teem in air.

No changes in the selection of raw materials or the melting procedures are needed.

INTRODUCTION

The internal quality of 50 kg steel ingots made in the PMRL foundry has been examined and the effects of melting, deoxidation and teeming procedures have been reviewed.

Part A of this report presents an evaluation of the foundry procedures and some suggestions for modifications. Part B is a more detailed account of measurements of product quality and experiments to improve deoxidation methods. Most of the work has been concerned with 50 kg heats but a few results from larger heats are also reported.

A. Review of Melting and Teeming Procedures

1. Melting and Alloying

The melting stock of continuously cast low-carbon steel billets has low sulphur, phosphorus and nitrogen contents and provides a good basis for PMRL melts. Recent checks by wet analysis (on three random samples from each billet type) gave the following results:

Element	Billet marks	
	White	Yellow
%S	0.0063	0.0092
%P	0.0059	0.011
%N	0.0017	0.0033

The billets contain numerous small glassy inclusions which probably consist mainly of manganese silicate but there is no evidence that these have any effect upon ingot quality, which depends upon the conditions of deoxidation and teeming.

The "argon cover" applied during the melting period cannot eliminate oxidation of the melt but probably restricts it. "Sparking" as the steel begins to melt is an effect of the induced currents at metal-to-metal contacts but oxidation of the pool can also cause a spray of droplets as a result of CO formation in the metal of low silicon content. Small additions of ferro-silicon should be made to keep this in check.

Manganese and the remaining ferro-silicon are added as soon as the charge has melted. The amount of slag is normally small but has been found

to contain as much as 35% MnO. It could contribute an unpredictable amount of manganese to the steel at the time of melt deoxidation if it is not removed.

The total oxygen content of a melt of 0.26% C steel after slag-off was about 150-200 ppm and was present as small suspended silicate globules. It is unlikely that they came directly from the billets because inclusions are continually being absorbed by the slag and onto the crucible walls while new ones form because the dissolved Si and Mn react with oxygen picked up from the air.

Alloying elements which oxidize slowly or not at all (e.g., Cr, Nb, Ni, Mo) are added at this point. Strongly oxidizable elements (e.g., Ti, Zr, V, B) are better added just before furnace-tapping or in the ladle.

2. Deoxidation

Aluminum is used as the main deoxidizing element for the following reasons:

a) the resulting total oxygen contents are the lowest that can be reached without special injection or vacuum treatments;
b) the practice is similar to that employed for industrial "killed steels" and gives a grain-refining effect in the end-product.

This element, when used alone, has some disadvantages:

i) the oxide, alumina, is precipitated as aggregates which are seen in polished sections as loose clusters of particles. Hot-rolling deforms these to stringers of particles which reduce the ductility and impact toughness of the rolled steel in the transverse and through-thickness directions;
ii) aluminum which is in solution at the time of furnace-tapping or ingot-teeming can react with air to form additional clusters of particles: higher concentrations of dissolved aluminum produce larger clusters.

The former practice of adding aluminum in the transfer ladle allowed very little time for the alumina clusters to separate out, particularly in the

50 kg melts and large clusters were commonly seen in polished sections from ingots. Experiments on modifying the deoxidation procedure are described in Part B of this report and ways of using either aluminum or the alloy "Hypercal" to give improved ingot quality are set out.

3. Control of Compositions

Spoon samples taken from the furnace and cast into ceramic ring-moulds for spectrographic analysis have given satisfactory results. Samples have also been taken with Geffner vacuum tubes. The 5.5 mm diam pins are suitable for combustion analysis of oxygen and nitrogen and can be cut into thin slices for wet analysis of soluble aluminum but are too small for spectrographic analysis. One commercial dip-sampler, giving a "lollipop" shape, was tested but its cardboard sheath caused too much boiling action in the crucible. No other forms of dip sampler have been found which are free from this problem and also give a sample suitable for spectrographic analysis or for drilling.

The emphasis of the work so far has been on the internal quality of ingots rather than on control of composition and no detailed study has been made of variations in composition within ingots. Sulphur printing of axial sections from two ingots (which contained 0.005 and 0.025% S) showed surprisingly little added concentration of sulphur in the central regions. Sulphur is an element which normally segregates very easily. It is therefore inferred that there are no other major variations in composition within these rapidly-solidifying ingots. Routine checks on ingot compositions have been made on corners of slices cut close to the ingot base.

4. Ingot-Teeming

A tap temperature of 1650°C for the 50 kg heats gave ingots of good internal structure with no piping and only minor microporosity in the central regions of a few ingots. In principle, different grades of steel need different teeming temperatures but the structure and quality of these rapidly-solidifying ingots are not sensitive to moderate variations. It is simpler to retain 1650°C as the standard temperature for furnace-tapping, allowing an extra 25 to 50° when a low-carbon steel is made or reducing the temperature when a highly alloyed grade such as stainless steel is to be cast.

For reasons to be given in Part B, a tundish has been made so that the 50 kg melts can be teemed without the need for a transfer ladle. The furnace is tapped directly into the tundish; as soon as this is full, the refractory-coated graphite stopper-rod is withdrawn and the liquid drains smoothly into the mould, which is first covered with plastic film and flushed out with argon. Here also the "standard" tapping temperature of 1650°C has been retained without adverse effects on ingot quality although a level at least 25° lower might be used in the absence of a ladle-transfer step.

B. Experiments on Deoxidation

1. Measurement of Ingot Quality

"Quality" is difficult to define and more difficult to measure. The most obvious factor is the quantity of oxide inclusions (alumina, aluminates, silicates) but even this cannot be determined accurately and reasonably quickly.

Analysis of small samples for oxygen by the vacuum-fusion method gave highly variable results for many ingots (see Table 4) and showed no consistent effects of sample location within an ingot. The determinations are believed to be accurate and it must be concluded that the oxygen contents of the compact 1 g samples were affected by the micro-segregation which exists in the coarse as-cast structure.

Standard charts used in the industrial "rating" methods (such as ASTM-E45) are designed for rolled steel and do not depict the undeformed alumina clusters. The 'D' scale of the E45 chart shows dispersed "globular oxides" but it was found difficult to match the wide range of particle sizes in a cast structure with the limited range depicted.

Visual counting of particles seen under the microscope is slow and calls for subjective judgements on the relative importance of particles of different sizes. Counts on a given specimen made on different days gave widely-differing results.

The Quantitative Television Microscope can objectively measure the area fraction of particles in a polished section. To scan 80 fields in one specimen takes 40 to 50 min but covers slightly less than 3 mm^2 in total. Nevertheless, some broad indications on particle-size distribution and oxide quantity have been provided. The data are given in Table 4 and are discussed in Section B5, below.

For the needs of most users of the ingots, the presence of clusters or "galaxies" of alumina particles is probably more important than the total quantity of oxides. A method has been devised for measuring cluster size and is described in the Appendix.

Results from a number of ingots showed that the "edge" specimens, which extended 15-20 mm from the ingot surface, contained smaller clusters than those found elsewhere but there were no consistent differences between those at centre and "half-radius" locations or at the top, middle and bottom locations in each ingot. For a quick measurement of ingot quality, a half-radius specimen from the middle slice is as nearly representative as a single specimen can be but for more complete comparisons, centre and half-radius specimens from top, middle and bottom slices should be examined. The test-work has shown that the range and mean values of readings from a set of six specimens is responsive to changes in deoxidation practice (Tables 1 and 2) and gives reasonably consistent values for similar deoxidation practices (Table 4).

2. Effects of the Timing of Aluminum Additions

In one 50 kg heat of 0.25% C steel, 0.06% Al was added to the melt and pin samples were taken with Geffner vacuum tubes at intervals of 1 to 1.5 min. Sufficient power was maintained to hold the metal temperature close to 1600°C.

Metallographic studies showed that the largest alumina clusters (e.g. of 8 area units) were formed not immediately after the addition but a little more than half a minute after it. Thereafter the sizes of clusters seen in the samples decreased to low values reaching 0.8 units after 4.25 min and 0.1 units after 5.5 min. The content of acid-soluble aluminum decreased with time and appeared to follow a first-order curve,

i.e., Rate of loss by oxidation at any instant
= concentration of soluble Al at that instant.

The total oxygen content of the melt was estimated to be over 200 ppm just after the aluminum had been added and decreased to 60 ppm or a little lower after about 3 min, after which it appeared to remain roughly constant (up to tapping after 6.5 min).

The heat was poured from a ladle and the alumina cluster-size in the ingot increased a little, to about 0.8 units but the oxygen content rose to roughly 100 ppm (Heat 1098, Table 4). As a check on the best holding time, heats were made with the same aluminum addition and intervals of 2, 4 or 6 min prior to tapping. The last of these gave the best results (Heat 1119, Table 4: see also Section 5 below).

Samples cut from ingots made after various Al additions and holding times showed that higher soluble Al contents at the time of furnace-tapping resulted in larger clusters in the ingot. Oxygen analyses however gave no clear picture on the total contents of oxide inclusions in relation to soluble % Al but gave higher results for ingots than for furnace samples because of oxygen pick-up during tapping and pouring.

A furnace addition of 0.06% Al followed by 5 to 6 min of holding at constant temperature was judged to give the greatest reduction in the cluster sizes of alumina particles in the ingot.

3. Use of the Tundish

In order to reduce the exposure of the steel to atmospheric oxidation, a tundish was constructed so that an ingot mould could be filled directly from the 50 kg furnace. As soon as the tundish had been filled with steel, the coated graphite stopper-rod was withdrawn and the liquid drained quietly into the mould, which had been covered with a plastic film and flushed with argon.

The results in Table 1 and summarized in Table 2 show that this reduced the cluster-size at high levels of soluble Al (i.e., after direct additions to tundish or ladle) but had little or no effect at low Al levels.

A direct addition of Al to the tundish produced alumina clusters which were, if anything, smaller than those produced by a furnace addition with 6 min of holding followed by ladle-pouring. The reason for the unexpectedly good results is not clear: it may be that in the very short time from addition to mould-filling the clusters had not developed to large sizes (see comment on the half-minute delay in Section B2, above).

In some metallurgical applications, the higher soluble Al content obtained by the tundish method would have some advantages for grain-size control.

4. Deoxidation with Hypercal

Hypercal contains 20% Al and 10% Ca, as well as Si and some Ba. This alloy has the effect of producing calcium aluminate as dispersed particles in place of the clustered alumina particles. It also replaces the usual sulphide, MnS, with CaS or a mixed compound (Ca,Mn)S which is deformed very little when the steel is hot-rolled.

A direct addition of Hypercal to the ladle reduced the average cluster size in the ingot from 15 to 2.8 units (Tables 1 and 2) but did not eliminate clusters. A furnace addition was not successful, probably because the calcium was vaporized or oxidized during the 4 min holding period.

An addition to the tundish of Hypercal wrapped in steel foil almost completely eliminated the alumina clusters. Out of 18 specimens from three ingots, seven showed no clusters and the largest cluster area measured in the others was only 0.7 units.

Partial deoxidation with 0.03 or 0.05% Al in the furnace, followed by a smaller Hypercal addition to the tundish did not eliminate all clusters in the ingots. Sections cut from rolled plate (21.5 mm thick) made from these ingots showed only a few oxide stringers and short sulphide streaks but these were sufficient to reduce the transverse impact toughness to about two-thirds of the longitudinal values (Table 3). Judging from results in the literature, this degree of anistropy is typical of untreated steels and further work will be needed to obtain more nearly equal results in the two directions. Tensile tests gave transverse ductility values which were over 90% of the longitudinal values.

5. Data from the Quantitative Television Microscope

The size distribution and total area of oxide particles were measured on 21 specimens, cut from eight ingots which represented the main lines of the deoxidation test-work. Table 4 shows the results together with comparative data from other heats made with closely similar deoxidation procedures.

There are wide differences among the results and it is difficult to relate these to the deoxidation procedures. Two heats which were treated almost identically gave different results (1098 to 1119) although the cluster-sizes were similar.

The average oxide content is higher for "top" samples than for "middle" and "bottom" levels but this might not be significant since the pattern is not consistent for each ingot. It is likely that the small areas surveyed in each specimen (about 3 mm^2) were too small to span the local variations in composition of the ingot structure; in other words, these, like the oxygen analyses represent only random local values within a pattern of microsegregation. This may also account for the lack of agreement or even of a uniform ratio between calculated and analyzed oxygen contents.

The tabulated results give the following broad indications:

a) Al addition to furnace + 5-6 min holding + tundish-teeming:
roughly 0.04-0.05% oxide, <6.0 ppm oxygen (analyzed);

b) Al added to tundish:
slightly higher oxide content (perhaps because of higher % Al soluble);

c) Hypercal added to tundish:
roughly 0.06% oxide, 60-80 ppm oxygen;

d) Al to furnace + Hypercal to tundish:
roughly 0.04% oxide, 50 ppm oxygen;

e) Hypercal, used alone, gave a larger number of particles (although these were well dispersed).

Other results (not shown) disclosed slightly larger particles in the ingot tops than at lower levels. The trend appeared in nearly all ingots (the others showing little variation) and there was also a less well-marked tendency for the volume fraction of oxides to be higher at the top.

The particles were much too small to rise by natural buoyancy during the short period while the ingot was still liquid and it is believed that dissolved oxygen was segregated as the ingot progressively solidified from the chilled based toward the hot-tap region.

The estimates of total oxygen content show a wide range of variations but it appears that levels below 50 ppm cannot be consistently attained when steel is melted and teemed in air.

6. A Note on Larger Melts

The principle of adding aluminum to the melt in the furnace, allowing time for the clusters to separate out is applicable to the melts of 150 to 225 kg made in the Brown-Boveri furnace but too little work has been done to establish the best quantity and holding time. The tundish cannot be used because of the high furnace lip and the need to fill several moulds but the longer time for filling, skimming and moving the large ladle may offset some of the effect of reoxidation during furnace-tapping.

Holding times of about 20 min, during which 0.11% Al was added in two portions, gave ingots with cluster sizes below 2.5 units (and at or below 1.2 units in 4 out of 5 heats). A basic slag, intended to absorb sulphur might have contributed by taking up inclusions and minimizing melt oxidation but the low residual contents of acid-soluble Al (<0.010%) at the time of furnace tapping were also important.

Another melt in which 0.06% Al was added 5 min before tap time had clusters of 1.5-3.7 units (2.4 average). The indication is that longer holding might be needed. Further experiments are to be made on this aspect and on the use of Hypercal in large melts.

CONCLUSIONS

a. The existing raw materials and the procedures for melting, sampling and temperature control are satisfactory. Changes to the deoxidation and pouring practices are recommended below.

b. Alumina clusters are the most important factor in the quality of ingots melted and poured in air.

c. Addition of aluminum to the furnace instead of to the ladle allowed time for the separation of the larger alumina clusters so that the liquid steel contained fewer inclusions and only small clusters after 4-5 min of holding.

d. The use of a special tundish allowed 50 kg melts to be poured directly into ingot moulds with minimal exposure to the air and reduced the size of alumina clusters in the ingot.

e. Hypercal added to the tundish at the time of furnace-tapping was very effective in dispersing the oxide inclusions and eliminating the clusters. Streak-like inclusions in plates rolled from the steel were few and short. These had very little detrimental effect on the transverse ductility but reduced the transverse toughness by about one-third.

f. There is no relation between cluster size and the volume fraction of oxide particles in the steel. Aluminum and Hypercal had about equal effects on the amount of oxides (measured as volume fraction or as total analyzed oxygen).

g. It does not appear to be practical to attain total oxygen contents below 50 ppm on a consistent basis in steel which is melted and poured in air.

h. Larger numbers of determinations of inclusion volume or total oxygen content should be made on each ingot, although the present arrangements do not facilitate this.

Recommendations for Deoxidation

1. For the smallest number and sizes of alumina clusters, add Hypercal to the tundish at the time of furnace-tapping (0.25% Hypercal, containing the equivalent of 0.05% Al).

2. For the lowest total oxygen content, add 0.06% Al to the furnace and hold for 5 to 6 min (not more) prior to tapping into the tundish.

3. If it is important to retain at least 0.02% soluble Al in the ingot, use Hypercal as in item 2 or add 0.06% Al directly to the tundish.

Table 1 - Effects of deoxidation treatments (100 lb/23 kg melts except in line 3)

Deoxidizing treatment	Teeming	Aluminum as deoxidizer							Hypercal as deoxidizer						
		Largest cluster areas			Total oxygen: ppm			Soluble Al%	Largest cluster areas			Total oxygen: ppm			Soluble Al%
		No.*,	range,	mean	No.,	range,	mean		No.,	range,	mean	No.,	range,	mean	
Deoxr. added to ladle	Ladle	2/14	0.8-56	15	2/14	15-80	42	.024, .034	1/6	0.8-5	2.8	1/2	86,97	92	.024, .030
Deoxr. to furnace 4-6' hold, then tap	Ladle	2/10	0.5-2.6	1.1 (6' holding)	2/8	39-130	70	.001	1/6	0.8-19	7.4 (4' holding)	1/2	63,94	78	.002
Deoxr. to 500 lb foe. about 20' hold	Ladle	5/10	0-2.4	0.8				.002, .011							
Deoxidizer added in tundish	Tundish	1/7	0.2-1.5	0.7	1/2	49,72	60	.044	3/18	0-0.7	0.2	3/12	19-101	65	.021, .030, .034
Deoxr. to furnace 5' holding, then tap	Tundish	3/18	0.2-3	1.2	3/12	25-69	52	.012, .023, .030							
Al to furnace, 5' hold, Hypercal to tundish	Tundish								3/15	0.3-2.5	0.75	3/7	40-101	55	.018

*No. of ingots tested/total No. of results (for soluble Al, one determination in each ingot)

Table 2 - Summary of cluster areas (averages)

	Aluminum		Hypercal	
	Ladle	Tundish	Ladle	Tundish
Direct addition	15	0.7	2.8	0.2
Furnace addition + 5-6 min hold	1.1	1.2	7.4	-
Addition to 500 lb furnace, 17-20' hold	0.8			

Table 3 - Tensile test results for rolled plate
21.5 mm thick, from 20 kg ingots
Deoxidation: Al in furnace, Hypercal in tundish

Property		Heat No. 1282		Heat No. 1283	
		Long'l	Transverse	Long'l	Transverse
Yield strength	MPa	443	508	436	452
U.T.S.	MPa	594	626	580	583
Red'n of area	%	60	58	61	56
Elongation	%	32	31	34	31
Notch impact tgh	J	80,82,85	58,59,63	96,98,102	61,63,65
Average		82	60	99	63
% of Long'l value		-	73	-	64

Table 4 - Summary of data from Quantitative Television Microscope and comparisons with other data

Heat No.	% Al	Deoxidation treatment	Q.T.M. readings					Calc'd	Other data				
			Average No. of particles	Inclusions Vol %					Analyzed oxygen ppm		Soluble Al	Alumina cluster area	
				T	M	B	Av.		Readings	Av.	%	Max.	Av.
1098	0.06	to F., 6 1/4' hold, ladle-poured	86	-	.026	.026	.026	62	85,127,130	(114?)	0.001	1.1	0.95
1119	0.06	to F., 6' hold, ladle-poured	146	.066	.055	-	.060	144	39,41,41,42	41	0.001	2.6	1.1
1245	0.06	to F., 5' hold, tundish-teemed	142	.046	.052	.020	.039	94	52,57,57,57	56	0.020	3	1.1
1354	0.06	to F., 5' hold, tundish-teemed	-	-	-	-	-	-	38,39	39	0.023	1.8	1.6
1356	0.05	to F., 5 1/4' hold, tundish teemed	-	-	-	-	-	-	30,54,54,56,56,59	53	0.012	2.4	1.1
1217	0.06	to tundish at tapping-time	86	.051	.048	.035	.044	106	49,72	(60?)	0.044	1.5	0.7
1186	0.05	as Hypercal, to ladle	217	.085	.078	.068	.077	140	86,97	92	.024, .030	5.0	2.8
1246	0.05	as Hypercal, to tundish at tap-time	263	.057	.061	.063	.061	98	34,69,81,82	67	0.030	0.5	0.1
1351	0.05	as Hypercal, to tundish at tap-time	-	-	-	-	-	-	19,57	(39?)	0.023	0.7	0.3
1352	0.04	as Hypercal, to tundish at tap-time	-	-	-	-	-	-	29,68,69,78,90,101	73	0.012	0.7	0.2
1256	0.05	to F., then-											
	0.025	as Hypercal to tundish	164	.059	.034	.062	.052	94	40,49,53,54	49	0.018	1.4	0.8
1283	0.04	to F., then-											
	0.03	as Hypercal to tundish	131	.052	-	.025	.038	67	45,101	(73?)	0.030	1.2	0.75

(F = furnace)

NOTES: QTM counts particles > 1 um d in 80 fields (500x) for each specimen

Calc'd [O] ppm: from density and composition of particles (approx. only for Hypercal-treated steel, as aluminate composition and density is variable)

Alumina cluster area: see the Appendix

APPENDIX - A METHOD FOR MEASURING THE SIZES OF ALUMINA CLUSTERS

A polished specimen of about 10 x 13 mm is scanned systematically at 100 magnifications. The dimensions of the largest cluster ("galaxy") are measured as follows:

Because the shapes are irregular, the longest dimension and the dimension perpendicular to it are measured (in micro-metres or microns = 0.001 mm) with an eyepiece graticule. Assuming these dimensions to be the axes of a lozenge shape, the area is L.W/2. To give manageable figures the result is divided by 1000: then the units are of 0.001 mm^2.

One unit might represent a measured size of 50 x 40 microns. After 6/1 rolling reduction (e.g., to a square billet section) the dimensions of a section of the same cluster would be about 300 x 16 microns or an apparent size (at 100x) of 30 x 1.6 mm. On the ASTM-E45 chart this would be rated at B2 which is acceptable for many industrial applications where transverse ductility and toughness are not critical factors.

Areas of 0.2 units or less usually include fewer than ten particles and scarcely qualify as "clusters".